

EXPRESS MAIL LABEL NUMBER EL822429368US

Form PTO-1390 REV.10-94)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEYS DOCKET NUMBER RN99013
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/elected office (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		U.S. APPLICATION NO. (IF KNOWN, SEE 3737 CFR 1.5) 09/890944 NOT APPLICABLE		
INTERNATIONAL APPLICATION NO. PCT/FR00/00371	INTERNATIONAL FILING DATE February 15, 2000	PRIORITY DATE CLAIMED February 17, 1999		
TITLE OF INVENTION USE OF FILM-FORMING TITANIUM DIOXIDE DISPERSIONS FOR CLEANING AND DISINFECTION SURFACES, FILM-FORMING TITANIUM DIOXIDE DISPERSIONS				
APPLICANT (S) FOR DO/EO/US: Eric AUBAY , Thierry CHOPIN , Cédric GEFFROY, Veronique GUILLOU, and CORINNE LEHAUT				
Applicant herewith submits the United States Designated/Elected Office (DO/EO/US) the following items and other information: 1 <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U S C 371 2 <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U S C 371 3 <input checked="" type="checkbox"/> This express request to begin national examination procedures (35 U S C 371) (f) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U S C 371 (b) and PCT Articles 22 and 39(1) 4 <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date 5 <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U S C 371(c)(2)) a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau). b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US) 6 <input checked="" type="checkbox"/> A translation of the International Application into English (35 U S C 371(c)(2)). 7 <input checked="" type="checkbox"/> A copy of the International Search Report (PCT/ISA/210) 8 <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U S C 371(c)(2)). a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau). b. <input type="checkbox"/> have been transmitted by the International Bureau c. <input type="checkbox"/> have not been made, however the time limit for making such amendments has NOT expired. d. <input checked="" type="checkbox"/> have not been made and will not be made. 9 <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U S C 371(c)(3)). 10 <input checked="" type="checkbox"/> A oath or declaration of the inventor(s) (35 U S C 371(c)(4)). (unsigned) 11 <input checked="" type="checkbox"/> A copy of the International Preliminary Examination Report (PCT/IPEA/409). 12 <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U S C 371(c)(5)). Items 13 to 18 below concern document(s) or information included: 13 <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98 14 <input type="checkbox"/> An assignment document for recording A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included 15 <input checked="" type="checkbox"/> A FIRST preliminary amendment 16 <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment 17 <input type="checkbox"/> A substitute specification 18 <input type="checkbox"/> A change of power of attorney and/or address letter 19 <input checked="" type="checkbox"/> Certificate of Mailing by Express Mail 20 <input checked="" type="checkbox"/> Other items or information 				
Forms PCT/IB/308 and 332, PCT/ISA/ 210 (English & French Version), PCT/IPEA/ 409, PCT/FR00/00371 as published				

US APPLICATION NO. IF KNOWN SEE 37 CFR 1.64 097890944		International Application No. PCT/FR00/00371	ATTORNEY DOCKET NUMBER RN99013																																																				
CALCULATIONS PTO ONLY																																																							
<p>21 The following fees are submitted:</p> <p>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</p> <p><input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1,482) nor International search fee (37 CFR 1.445(a)(2) paid to USPTO and International Search Report not prepared by the EPO or JPO..... \$1,000.00</p> <p><input checked="" type="checkbox"/> International preliminary examination fee not paid to USPTO but International Search Report prepared by the EPO or JPO..... \$ 860.00</p> <p><input type="checkbox"/> International preliminary examination fee (37 CFR 1,482) not paid to USPTO but international search (37 CFR 1,4445 (a)(2)) fee paid to USPTO..... \$ 710.00</p> <p><input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1,482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$ 690.00</p> <p><input type="checkbox"/> International preliminary examination fee paid to USPTO (37 CFR 1,482) and all claims satisfied provisions of PCT Article 33(2)-(4) \$ 100.00</p> <p style="text-align: center;">ENTER APPROPRIATE BASIC FEE AMOUNT =</p>																																																							
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<p>a <input type="checkbox"/> A check in the amount of \$ _____ to cover the above fees is enclosed</p> <p>b <input checked="" type="checkbox"/> Please charge my Deposit Account No. 18-1171 in the amount of \$1,490.00 to cover the above fees.</p> <p>c <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 18-1171 A duplicate copy of this sheet is enclosed.</p>																																																							
<p>NOTE: Where an appropriate time limit under 37 CFR 1,494 or 1,495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.</p>																																																							
<p><u>SEND ALL CORRESPONDENCE TO:</u> John Daniel WOOD</p>																																																							
<p><u>RHODIA INC.</u> 259 Prospect Plains Road CN 7500 Cranbury, NJ 08512</p>																																																							
<p><u>August 6 , 2001</u></p>																																																							
<p><u>REGISTRATION NUMBER</u></p>																																																							

Case RN99013

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

**In the Application of: Eric AUBAY , Thierry CHOPIN , Cédric GEFFROY,
Veronique GUILLOU, and CORINNE LEHAUT**

National Phase of PCT/FR00/00371

Examiner: To be assigned

International Filing Date : February 15, 2000

Serial No: To be assigned

Art Unit: To be assigned

Filing Date: To be assigned

**For: USE OF FILM-FORMING TITANIUM DIOXIDE DISPERSIONS FOR
CLEANING AND DISINFECTING SURFACES, FILM-FORMING TITANIUM
DIOXIDE DISPERSIONS**

Assistant Commissioner for Patents

Washington, D.C. 20231

PRELIMINARY AMENDMENT

Dear Sir:

Prior to calculation of filing fee, please enter the following amendment in the specification and claims:

In the specification:

Page 1, just after the title, please add the new following amendment:

This application is an application under 35 U.S.C. Section 371 of International Application Number PCT/FR00/00371 filed on February 15, 2000.

In the claims:

Please cancel claims 1 to 33 and replace them with the new following claims 34 to 88 :

34) (New) A process for cleaning or disinfecting surfaces exposed to light, comprising the step of depositing a film of titanium dioxide on said surfaces with a film-forming dispersion comprising a continuous phase, said titanium dioxide

being in the form of elementary particles whose size is less than 100 nm, and whose specific surface area is greater than 150 m²/g, the continuous phase of said dispersion comprising water or at least one alcohol whose boiling point is less than 120°C, and said dispersion having, when it comprises water, a pH different by at least 1 unit, from the value of the isoelectric point of titanium dioxide in said dispersion.

35) (New) A process according to claim 34), wherein the size of the particles is less than 70 nm, the specific surface area of the particles is greater than 200 m²/g, the boiling point of the alcohol is less than or equal to 100°C, and the pH is different by at least 2 units, from the value of the isoelectric point of titanium dioxide in the dispersion.

36) (New) A process according to claim 34), wherein the size of the particles is from 20 to 60 nm, and the specific surface area is from from 200 to 300 m²/g.

37) (New) A process according to claim 34) wherein the particles of titanium dioxide are anatase.

38) (New) A process according to claim 34) wherein the dispersion comprises from 0.01 to 15% of its weight of titanium dioxide.

39) (New) A process according to claim 34) wherein the dispersion comprises from 0.1 to 10% of its weight of titanium dioxide.

40) (New) A process according to claim 34) wherein the continuous phase comprises an aliphatic monoalcohol whose boiling point is less than 100°C.

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41) (New) A process according to claim 34) wherein the continuous phase comprises water.

42) (New) A process according to claim 41) wherein the continuous phase comprises from 50 to 100% water.

43) (New) A process according to claim 42) wherein the continuous phase comprises from 70 to 99.9% water.

44) (New) A process according to claim 41) wherein the dispersion has a pH of from 0 to 14.

45) (New) A process according to claim 42) wherein the dispersion has a pH of from 2 to 14.

46) (New) A process according to claim 44), wherein the pH of the dispersion is less than 4 or more than 8, when the dispersion does not comprise an additive modifying the isoelectric point of titanium dioxide.

47) (New) A process according to claim 46), wherein the pH of the dispersion is from 0 to 3, or from 9 to 14, when the dispersion does not comprise an additive modifying the isoelectric point of titanium dioxide.

48) (New) A process according to claim 34) wherein the dispersion further comprises at least one film-forming organic or organosiloxane polymer.

49) (New) A process according to claim 48) wherein the dispersion comprises from 0.005 to 15% of the film-forming polymer.

50) (New) A process according to claim 49) wherein the dispersion comprises from 0.01 to 10% of the film-forming polymer.

51) (New) A process according to claim 48) wherein the film-forming polymer is selected from the group consisting of:

- terephthalic oligoesters or copolyesters, optionally sulfonated,
- polyoxyalkylenated polymers which carry anionic functions,
- cationic polymers,
- amphoteric polymers,
- polyalkoxylated polydimethylsiloxanes, and
- film-forming polymers derived from ethylenically unsaturated monomers polymerizable by radical means.

52) (New) A process according to claim 51), wherein the film-forming polymer interacts with the surface of the titanium dioxide particles by electrostatic bonding.

53) (New) A process according to claim 52) wherein the polymer is selected from the group consisting of:

- a sulfonated terephthalic oligoester or copolyester,
- a polyoxyalkylenated polymer which carries anionic functions,
- a cationic polymer additionally exhibiting bacteriostatic properties,
- a cationic homopolymer or copolymer derived from at least one cationic monomer comprising an ethylenic unsaturation, optionally in a mixture with at least one nonionic monomer, and

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- an amphoteric copolymer derived from at least one cationic monomer comprising an ethylenic unsaturation, and at least one anionic monomer comprising an ethylenic unsaturation, optionally in a mixture with at least one nonionic monomer.

54) (New) A process according to claim 52) wherein the film-forming polymer is selected from the group consisting of:

- copolymers of acrylic acid or methacrylic acid and polyethylene glycol acrylate or methacrylate having a polyoxyethylene mass of from 500 to 10 000,
- quaternary ammonium ionenes,
- cationic polymers derived from epichlorohydrin and dimethylamine, and
- cationic polymers derived from epichlorohydrin and imidazole

55) (New) A process according to claim 53) wherein the cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

- aminoacryloyl or -acryloyloxy monomers,
- N,N-dialkyldiallylamine monomers
- polyquaternary monomers.

56) (New) A process according to claim 55) wherein the cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

trimethylaminopropyl methacrylate chloride, trimethylaminoethylacrylamide or trimethylaminomethacrylamide chloride or bromide, trimethylaminobutylacrylamide or trimethylaminobutylmethacrylamide methyl sulfate, trimethylaminopropyl-

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methacrylamide methyl sulfate (MES), (3-methacrylamidopropyl)trimethylammonium chloride (MAPTAC), (3-acrylamidopropyl)trimethylammonium chloride (APTAC), methacryloyloxyethyl trimethylammonium chloride or methyl sulfate, acryloyloxyethyltrimethylammonium chloride, 1-ethyl-2-vinylpyridinium bromide, 1-ethyl-2-vinylpyridinium chloride, 1-ethyl-2-vinylpyridinium bromide methyl sulfate, N,N-dimethyldiallylammonium chloride (DADMAC), dimethylaminopropylmethacrylamide chloride, and N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT)

57) (New) A process according to claim 53) wherein the anionic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

- acrylic, methacrylic, fumaric, maleic, itaconic, N-methacroylalanine, or N-acryloylhydroxyglycine acids or anhydrides, or their water-soluble salts, and
- water-soluble sulfonated or phosphonated ethylenically unsaturated monomers.

58) (New) A process according to claim 57) wherein the anionic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

sulfopropyl acrylate, its water-soluble salts, water-soluble styrenesulfonates, vinylsulfonic acid, its water-soluble salts, vinylphosphonic acid, and its water-soluble salts.

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59) (New) A process according to claim 53) wherein the nonionic monomer is selected from the group consisting of acrylamide, N-isopropylacrylamide, N,N-dimethylacrylamide, dimethylaminoethylmethacrylate (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohols, alkyl acrylates or methacrylates, hydroxyalkyl acrylates or methacrylates, and polyoxyalkylene glycol acrylates or methacrylates.

60) (New) The use as claimed in claim 53) wherein the cationic or amphoteric polymer derived from at least one cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

- a DIQUAT homopolymer;
- a DADMAC homopolymer;
- a MAPTAC/acrylic or methacrylic acid; a DIQUAT/acrylic or methacrylic acid; a DADMAC/acrylic or methacrylic acid copolymer;
- a MES/acrylic or methacrylic acid/DMAEMA copolymer;
- a MAPTAC/acrylic acid/acrylamide; a MAPTAC/maleic anhydride/acrylamide; a MAPTAC/vinylsulfonic acid/acrylamide copolymer;
- a DADMAC/acrylic acid/acrylamide; a DADMAC/maleic anhydride/acrylamide; a DADMAC/vinylsulfonic acid/acrylamide copolymer;
- a DIQUAT/acrylic acid/acrylamide; a DIQUAT/maleic anhydride/acrylamide; and a DIQUAT/vinylsulfonic acid/acrylamide copolymer;

copolymers having a ratio of the total number of anionic charges to the total number of cationic charges of from 95/5 to 5/95,

61) (New) A process according to claim 60) wherein the ratio is from 90/10 to 10/90.

62) (New) A process according to claim 53) wherein the dispersion comprises from 0.01 to 2% by weight of the film-forming polymer interacting with the surface of the titanium dioxide particles by electrostatic bonding.

63) (New) A process according to claim 52), wherein the dispersion comprises water and has a pH of from 4 to 9.

64) (New) A process according to claim 34) wherein the amount of titanium dioxide deposited on the surfaces is from 0.01 to 10 g of titanium dioxide per m² of the surface to be treated.

65) (New) A process according to claim 64) wherein the amount of titanium dioxide deposited on the surfaces is from 0.05 to 5 g of titanium dioxide per m² of the surface to be treated.

66) (New) A film-forming dispersion comprising:

- from 0.01 to 15% of its weight of titanium dioxide in the form of elementary particles whose size is less than 100 nm, and whose specific surface area is greater than 150 m²/g,
- from 0.005 to 15% of its weight of at least one film-forming polyalkoxylated organosiloxane or organic polymer, and
- a continuous phase of said dispersion comprising water or at least one alcohol whose boiling point is less than 120°C, and having, when it comprises water, a pH different

by at least 1 unit, from the value of the isoelectric point of titanium dioxide in said dispersion.

67) (New) A film-forming dispersion according to claim 64, wherein the size of the particles is less than 70 nm, the specific surface area of the particles is greater than 200 m²/g, the boiling point of the alcohol is less than or equal to 100°C, and the pH is different by at least 2 unit, from the value of the isoelectric point of titanium dioxide in the dispersion.

68) (New) A film-forming dispersion according to claim 66), characterized in that the titanium dioxide is in the form of elementary particles whose size is of the order of from 20 to 60 nm and whose specific surface area is of the order of from 200 to 300 m²/g.

69) (New) A film-forming dispersion according to claim 66), wherein the particles of titanium dioxide are anatase.

70) (New) A film-forming dispersion according to claim 66), wherein the continuous phase comprises an aliphatic monoalcohol whose boiling point is less than 100°C.

71) (New) A film-forming dispersion according to claim 66), wherein the continuous phase comprises water.

72) (New) A film-forming dispersion according to claim 71) wherein the continuous phase comprises from 50 to 100% water.

73) (New) A film-forming dispersion according to claim 72) wherein the continuous phase comprises from 70 to 99.9% water.

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74) (New) A film-forming dispersion according to claim 71)

wherein the dispersion has a pH of from 0 to 14.

75) (New) A film-forming dispersion according to claim 74)

wherein the dispersion has a pH of from 2 to 14.

76) (New) A film-forming dispersion according to claim 66),

wherein the film-forming polymer is selected from the group consisting of:

- terephthalic oligoesters or copolyesters, optionally sulfonated,
- polyoxyalkylenated polymers which carry anionic functions,
- cationic polymers,
- amphoteric polymers,
- polyalkoxylated polydimethylsiloxanes, and
- film-forming polymers derived from ethylenically unsaturated monomers polymerizable by radical means.

77) (New) A film-forming dispersion according to claim 76)

wherein the film-forming polymer interacts with the surface of the titanium dioxide particles by electrostatic bonding.

78) (New) A film-forming dispersion according to claim 77)

wherein the polymer is selected from the group consisting of:

- a sulfonated terephthalic oligoester or copolyester,
- a polyoxyalkylenated polymer which carries anionic functions,
- a cationic polymer additionally exhibiting bacteriostatic properties,

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- a cationic homopolymer or copolymer derived from at least one cationic monomer comprising an ethylenic unsaturation, optionally in a mixture with at least one nonionic monomer, and
- an amphoteric copolymer derived from at least one cationic monomer comprising an ethylenic unsaturation, and at least one anionic monomer comprising an ethylenic unsaturation, optionally in a mixture with at least one nonionic monomer.

79) (New) A film-forming dispersion according to claim 77)

wherein the film-forming polymer is selected from the group consisting of:

- copolymers of acrylic acid or methacrylic acid and polyethylene glycol acrylate or methacrylate having a polyoxyethylene mass of from 500 to 10 000,
- quaternary ammonium ionenes,
- cationic polymers derived from epichlorohydrin and dimethylamine, and
- cationic polymers derived from epichlorohydrin and imidazole

80) (New) A film-forming dispersion according to claim 78)

wherein the cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

- aminoacryloyl or -acryloyloxy monomers,
- N,N-dialkyldiallylamine monomers
- polyquaternary monomers.

81) (New) A film-forming dispersion according to claim 80)

wherein the cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

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trimethylaminopropyl methacrylate chloride, trimethylaminoethylacrylamide or
trimethylaminomethacrylamide chloride or bromide, trimethylaminobutylacrylamide
or trimethylaminobutylmethacrylamide methyl sulfate, trimethylaminopropyl-
methacrylamide methyl sulfate (MES), (3-methacrylamidopropyl)trimethylammonium
chloride (MAPTAC), (3-acrylamidopropyl)trimethylammonium chloride (APTAC),
methacryloyloxyethyl trimethylammonium chloride or methyl sulfate,
acryloyloxyethyltrimethylammonium chloride,
1-ethyl-2-vinylpyridinium bromide, 1-ethyl-2-vinylpyridinium chloride, 1-ethyl-2-
vinylpyridinium bromide methyl sulfate,
N,N-dimethyldiallylammonium chloride (DADMAC),
dimethylaminopropylmethacrylamide chloride, and
N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT)

82) (New) A film-forming dispersion according to claim 78)

wherein the anionic monomer comprising an ethylenic unsaturation is selected from
the group consisting of:

- acrylic, methacrylic, fumaric, maleic, itaconic, N-methacroylalanine, or
N-acryloylhydroxyglycine acids or anhydrides, or their water-soluble salts, and
- water-soluble sulfonated or phosphonated ethylenically unsaturated monomers.

83) (New) A film-forming dispersion according to claim 82)

wherein the anionic monomer comprising an ethylenic unsaturation is selected from
the group consisting of:

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sulfopropyl acrylate, its water-soluble salts, water-soluble styrenesulfonates, vinylsulfonic acid, its water-soluble salts, vinylphosphonic acid, and its water-soluble salts.

84) (New) A film-forming dispersion according to claim 78)

wherein the nonionic monomer is selected from the group consisting of acrylamide, N-isopropylacrylamide, N,N-dimethylacrylamide, dimethylaminoethylmethacrylate (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohols, alkyl acrylates or methacrylates, hydroxyalkyl acrylates or methacrylates, and polyoxyalkylene glycol acrylates or methacrylates.

85) (New) A film-forming dispersion according to claim 78)

wherein the cationic or amphoteric polymer derived from at least one cationic monomer comprising an ethylenic unsaturation is selected from the group consisting of:

- a DIQUAT homopolymer;
- a DADMAC homopolymer;
- a MAPTAC/acrylic or methacrylic acid; a DIQUAT/acrylic or methacrylic acid; a DADMAC/acrylic or methacrylic acid copolymer;
- a MES/acrylic or methacrylic acid/DMAEMA copolymer;
- a MAPTAC/acrylic acid/acrylamide; a MAPTAC/maleic anhydride/acrylamide; a MAPTAC/vinylsulfonic acid/acrylamide copolymer;
- a DADMAC/acrylic acid/acrylamide; a DADMAC/maleic anhydride/acrylamide; a DADMAC/vinylsulfonic acid/acrylamide copolymer;

- a DIQUAT/acrylic acid/acrylamide; a DIQUAT/maleic anhydride/acrylamide; and
a DIQUAT/vinylsulfonic acid/acrylamide copolymer;
copolymers having a ratio of the total number of anionic charges to the total number of
cationic charges of from 95/5 to 5/95,

86) (New) A film-forming dispersion according to claim 85)

wherein the ratio is from 90/10 to 10/90.

87) (New) A film-forming dispersion according to claim 78),

wherein the dispersion comprises from 0.01 to 2% by weight of the film-forming
polymer interacting with the surface of the titanium dioxide particles by electrostatic
bonding.

88) (New) A film-forming dispersion according to claim 77),

wherein the dispersion comprises water and has a pH of from 4 to 9.

REMARKS

The preliminary amendments are filed to comply with the claims structure and
wording according to the United States Patent law. It is asserted that these
amendments do not add new matter. Support for these amendments can be found in
the specification and claims as originally filed.

New claims find basis as mentioned in the chart below:

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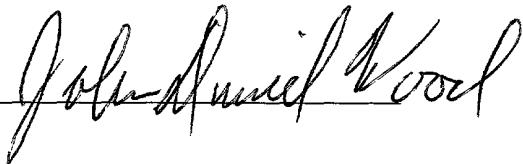
New Claims	Basis	
	In the claims as filed	In the specification
34, 35	1	“film forming” on page 1, lines 19-20
36	2	
37	3	
38, 39	4	
40	5	
41	6	
42, 43	7	
44, 45	8	
46, 47	9	
48	10	
49, 50	11	
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53-59	14	
60, 61	15	
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64, 65	32	
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72, 73	23	
74, 75	24	
76	25	
77	26	
78-84	27	
85, 86	28	
87	29	
88	30	

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Preliminary Amendment
PCT/FR00/00371

Entry of the amendments is respectfully requested.

Respectfully submitted,

By:



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USE OF FILM-FORMING TITANIUM DIOXIDE DISPERSIONS FOR
CLEANING AND DISINFECTION SURFACES, FILM-FORMING
TITANIUM DIOXIDE DISPERSIONS

The present invention relates to the use of a
5 film-forming dispersion of titanium dioxide
nanoparticles for cleaning and/or disinfecting surfaces
exposed to light. The invention also relates to a
process for cleaning and/or disinfecting surfaces
exposed to light by depositing on said surfaces a film
10 of titanium dioxide nanoparticles, and to titanium
dioxide nanoparticle dispersions whose film-forming
properties are improved by the presence of a film-
forming polymer.

The use of titanium dioxide nanoparticles as
15 a bactericidal and photooxidizing agent in detergent
compositions for the washing of laundry or of surfaces
was described by the Applicant in its French patent
application No. 95 00821 of January 25, 1996.

The Applicant has observed that titanium
20 dioxide nanoparticle dispersions are film-forming.

The object of the present invention is to
utilize the photooxidizing properties of titanium
dioxide nanoparticles and their film-forming nature to
clean and/or disinfect surfaces on said surfaces of an
25 impermanent film of titanium dioxide capable under
light of generating free radicals which disinfect the
surfaces and oxidize the soiling.

The creation of this film makes it possible

- to clean (remove soiling)
 - to control or even suppress bacterial proliferation between two operations of disinfecting by conventional means;
- 5 - to reduce the frequency of cleaning or disinfection and/or the quantity of cleaning or disinfecting products presently used.

The film formed is then progressively removed by the subsequent cleaning steps. The target fields of

10 application may be very varied, such as the cleaning or disinfecting of hard surfaces encountered in the food industry, kitchens, bathrooms, washrooms, hospitals, glazing, facades, etc, and also the breakdown and/or removal of heavy hydrocarbons deposited on surfaces,

15 particularly following accidental pollution (for example beaches, rocks, equipment, plants, etc).

The invention first provides for the use of a titanium dioxide dispersion for cleaning and/or disinfecting surfaces exposed to light by deposition of

20 a film of titanium dioxide on said surfaces, said titanium dioxide being in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is greater than 150 m²/g, preferably greater than 200 m²/g,

25 the continuous phase of said dispersion comprising water and/or at least one alcohol whose boiling point is less than 120°C, preferably less than or equal to 100°C, said dispersion having, when it comprises water,

a pH different by at least 1 unit, preferably by at least 2 units, from the value of the isoelectric point of titanium dioxide in said dispersion.

The pH of the dispersion comprising water is
5 of course the pH of the continuous phase.

The titanium dioxide employed is very particularly in the form of elementary particles whose size is of the order of from 20 to 60 nm and whose specific surface area is of the order of from 200 to
10 300 m²/g.

The specific surface area given is a BET surface. By BET surface is meant the specific surface area determined by nitrogen adsorption in accordance with the standard ASTM D 3663-78, based on the Brunauer
15 - Emmett - Teller method described in the journal "The Journal of the american Society", 60, 309 (1938). The size of the elementary particles of titanium dioxide according to the invention is measured by transmission electron microscopy (TEM) .

20 The nature of the elementary particles of titanium dioxide is preferably anatase (isoelectric point between 5.5 and 6 for pure anatase).

Within said dispersion, said elementary particles may be present in the form of both aggregates and
25 elementary particles.

For effective implementation of the invention, said dispersion may comprise in the order of from 0,01 to 15% of its weight, preferably in the order

of from 0.1 to 10% of its weight, of titanium dioxide.

Among the alcohols which may constitute or be present in the continuous phase, mention may be made in particular of aliphatic monoalcohols whose boiling point is less than 100°C such as ethanol, isopropanol, etc.

When the continuous phase consists of a water/alcohol(s) mixture whose boiling point is less than 120°C, the ratio between the water and the alcohol or alcohols is arbitrary.

A high boiling point alcohol (in particular a diol such as ethylene glycol) may, however, be present in the continuous phase, but may not represent more than 10% of the weight of said phase.

15 Preferentially, the continuous phase
comprises water, preferably from 50 to 100%, preferably
from 70 to 99.9% of its weight of water.

When the continuous phase comprises water, the pH values favorable to effective implementation of the invention may range from 0 to 14, preferably from 2 to 14, and are a function of other additives which may be present in the dispersion and capable of modifying the isoelectric point of the titanium dioxide.

When no additive capable of modifying the isoelectric point of titanium dioxide is present, the pH values favorable to effective implementation of the invention are situated below 4 or above 8; preferentially, the pH of the dispersion may range from 0 to 3 approximately

or from 9 to 14 approximately.

If necessary, pH regulators may be present; mention may be made of mineral acids or organic acids such as hydrochloric, nitric, sulfuric, phosphoric, 5 citric, glutaric, adipic and succinic acids, etc., and bases such as aqueous ammonia, alkali metal hydroxides, sodium carbonate, and triethanolamine, etc.

Said dispersion may further comprise other additives which are soluble or dispersible in the 10 continuous phase, said additives possibly promoting its stability, wettability, augmenting its film-forming nature, its biocidal nature, or providing other supplementary properties.

Examples of additives which may be mentioned 15 include:

- nonionic surfactants promoting wettability, of the polyoxyethylenated C₆-C₁₂ alkylphenol type, polyoxypropylenated and/or polyoxyethylenated C₈-C₂₂ aliphatic alcohol type, ethylene oxide-propylene oxide 20 block copolymers, optionally polyoxyethylenated carboxylic amides, etc,
- anionic or amphoteric surfactants as dispersants, of the alkali metal soap type (alkali metal salts of C₈-C₂₄ fatty acids), alkali metal sulfonates (C₈-C₁₃ 25 alkylbenzene sulfonates, C₁₂-C₁₆ alkylsulfonates), sulfated and oxyethylenated C₆-C₁₆ fatty alcohols, sulfated and oxyethylenated C₈-C₁₃ alkylphenols type, alkali metal sulfosuccinates (C₁₂-C₁₆ alkylsulfo-

succinates) etc, betaines, etc

- biocides or bacteriostatics capable of improving the biocidal nature of the dispersions, especially in the case of inadequate lighting, such as cationic

5 surfactants (alkyldimethylammonium halides, etc), quaternary ammonium or phosphonium halide biocides, glycine-derivative amphoteric biocides, phenolic biocides, chlorhexidine-derivative biocides, hypochlorites, quaternary polyammonium film-forming

10 polymers or biocides, etc

- film-forming antisoiling agents such as optionally sulfonated terephthalic polyesters, etc

- other film-forming homopolymers or copolymers such as those derived from monomers containing ethylenic

15 unsaturation, especially cationic or amphoteric homopolymers or copolymers derived from cationic monomers containing ethylenic unsaturation

- film-forming polyoxyalkylene polymers which carry anionic functions

20 - film-forming polymers which impart brightness and oleophobicity, such as polyalkoxylated organopolysiloxanes, etc.

- fragrances

- dyes, etc.

25 These various additives may be present in a proportion of from 0 to 15% by weight of said dispersion,

One particular embodiment of the invention consists in improving the film-forming nature of the

titanium dioxide nanoparticles by the presence within the dispersion of at least one organic or organosiloxane polymer of film-forming nature which may further impart one or more supplementary properties to
5 said dispersion.

The amount of polymer that may be present may represent in the order of from 0.005 to 15%, preferably in the order of from 0.01 to 10% of the weight of said dispersion.

10 Among the film-forming organic or organosiloxane polymers that may be employed, very particular mention may be made of:

- terephthalic polyesters additionally exhibiting antisoiling properties, such as
- 15 • polyester copolymers based on ethylene terephthalate and/or propylene terephthalate and polyoxyethylene terephthalate units (US-A-3 959 230, US-A-3 893 929, US-A-4 116 896, US-A-4 702 857, US-A-4 770 666);
- sulfonated polyester oligomers obtained by
- 20 sulfonating an ethoxylated allyl alcohol-derived oligomer, dimethyl terephthalate and 1,2-propylenediol (US-A-4 968 451)
- polyester copolymers based on propylene terephthalate and polyoxyethylene terephthalate units and terminated
- 25 with ethyl or methyl units (US-A-4 711 730) or polyester oligomers terminated with alkylpolyethoxy groups (US-A-4 702 857) or anionic sulfopolyethoxy groups (US-A-4 721 580) or sulfoaroyl groups (US-A-4 877 896)

- polyester-polyurethanes obtained by reacting a polyester obtained from adipic acid and/or terephthalic acid and/or sulfoisophthalic acid and a diol with a prepolymer containing terminal isocyanate groups
- 5 obtained from a polyoxyethylene glycol and a diisocyanate (FR-A-2 334 698)
- sulfonated polyester oligomers obtained by condensing isophthalic acid, dimethyl sulfosuccinate and diethylene glycol (FR-A-2 236 926)
- 10 • polyester copolymers derived from dimethyl terephthalate, isophthalic acid, dimethyl sulfoisophthalate and ethylene glycol (EP-A-540374)
- cationic polymers further exhibiting bacteriostatic properties, such as
- 15 • the quaternary ammonium ionenes described in US-A-4,157,388 (MIRAPOL A-15 or POLYQUATERNIUM-2 from Rhodia)
- cationic polymers derived from epichlorohydrin and dimethylamine and those derived from epichlorohydrin
- 20 and imidazole, such as GLOKILL PQ and ELC from Rhodia
- film-forming cationic or amphoteric homopolymers or copolymers derived from cationic monomers containing ethylenic unsaturation (described in more detail below)
- film-forming polyoxyalkylene polymers which carry
- 25 anionic functions, such as copolymers of acrylic acid and/or methacrylic acid and polyethylene glycol acrylate and/or methacrylate whose polyoxyethylene unit has a mass of the order of from 500 to 10 000

- polyalkoxylated polydimethylsiloxanes which impart brightness and oleophobicity, such as SILICONE COPOLYOL 10646 from Rhodia
 - other film-forming polymers derived from
- 5 ethylenically unsaturated monomers polymerizable by radical means, such as (meth)acrylic acid, C1-C4 alkyl (meth)acrylates, styrene, butadiene, etc.

One very particularly advantageous embodiment of the invention consists in the use of titanium dioxide dispersions further comprising a film-forming organic polymer which is preferably hydrophilic and is capable of interacting with the surface of the titanium dioxide particles, preferably by electrostatic bonding.

Said dispersions are preferably aqueous and

15 have a pH of the order of from 4 to 9.

They may comprise in the order of from 0.005 to 10%, preferably from 0.01 to 5%, very particularly from 0.01 to 2% by weight of film-forming organic polymer.

20 Among the film-forming polymers which may be present, mention may be made of:

- * the sulfonated terephthalic copolymers or oligoesters already mentioned above
- * film-forming polyoxyalkylene polymers which carry

25 anionic functions, such as copolymers of acrylic acid and/or methacrylic acid and polyethylene glycol acrylate and/or methacrylate whose polyoxyethylene unit has a mass of the order of from 500 to 10 000

- * film-forming cationic polymers additionally exhibiting bacteriostatic properties, such as
- the quaternary ammonium ionenes described in US-A-4,157,388 (MIRAPOL A-15 or POLYQUATERNIUM-2 from Rhodia)
 - cationic polymers derived from epichlorohydrin and dimethylamine and those derived from epichlorohydrin and imidazole, such as GLOKILL PQ and ELC from Rhodia
- * film-forming cationic or amphoteric homopolymers or copolymers derived from cationic monomers containing ethylenic unsaturation.

By way of example of cationic film-forming homopolymers or copolymers derived from cationic monomers containing ethylenic unsaturation, mention may be made from those derived from at least one of the following cationic monomers

- * aminoacryloyl or -acryloyloxy monomers such as trimethylaminopropyl methacrylate chloride, trimethylaminoethylacrylamide or -methacrylamide chloride or bromide, trimethylaminobutylacrylamide or -methacrylamide methyl sulfate, trimethylaminopropyl-methacrylamide methyl sulfate (MES), (3-methacrylamidopropyl)trimethylammonium chloride (MAPTAC), (3-acrylamidopropyl)trimethylammonium chloride (APTAC), methacryloyloxyethyltrimethylammonium chloride, acryloyloxyethyltrimethylammonium chloride;
- * 1-ethyl-2-vinylpyridinium bromide, chloride or methyl

sulfate;

* N,N-dialkyldiallylamine monomers such as

N,N-dimethyldiallylammonium chloride (DADMAC) ;

* polyquaternary monomers such as

5 dimethylaminopropylmethacrylamide chloride,

N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT),

etc;

optionally in a mixture with at least one nonionic monomer such as acrylamide, N-isopropylacrylamide,

10 N,N-dimethylacrylamide, dimethylaminoethylmethacrylate (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohol, alkyl or hydroxyalkyl acrylates or methacrylates, polyoxyalkylene glycol acrylates or methacrylates, etc.

15 Very particular mention may be made of DIQUAT homopolymers, such as MIRAPOL CLASTA from Rhodia, and DADMAC homopolymers, such as MIRAPOL 100 from Rhodia.

By way of example of amphoteric film-forming copolymers derived from cationic monomers containing

20 ethylenic unsaturation, mention may be made of those derived from at least one of the abovementioned cationic monomers and at least one anionic monomer such as

* acrylic, methacrylic, fumaric, maleic, itaconic,

25 N-methacroylalanine, N-acryloylhydroxyglycine, etc, acids or anhydrides, or their water-soluble salts;

* water-soluble sulfonated or phosphonated

ethylenically unsaturated monomers, such as sulfopropyl

acrylate or its water-soluble salts, water-soluble styrene sulfonates, vinylsulfonic acid and its water-soluble salts or vinylphosphonic acid and its water-soluble salts, etc;

- 5 optionally in a mixture of at least one nonionic monomer such as acrylamide, N-isopropylacrylamide, N,N-dimethylacrylamide, dimethylaminoethylmethacrylate (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohol, alkyl or hydroxyalkyl acrylates or
10 methacrylates, polyoxyalkylene glycol acrylates or methacrylates, etc.

Very particular mention may be made of the following copolymers or terpolymers:

- * MAPTAC/acrylic or methacrylic acid; DIQUAT/acrylic or
15 methacrylic acid; DADMAC/acrylic or methacrylic acid;
* MES/acrylic or methacrylic acid/DMAEMA;
* MAPTAC/acrylic acid/acrylamide; MAPTAC/maleic anhydride/acrylamide; MAPTAC/vinylsulfonic acid/acrylamide;
20 * DADMAC/acrylic acid/acrylamide; DADMAC/maleic anhydride/acrylamide; DADMAC/vinylsulfonic acid/acrylamide;
* DIQUAT/acrylic acid/acrylamide; DIQUAT/maleic anhydride/acrylamide; DIQUAT/vinylsulfonic
25 acid/acrylamide;

with a ratio of the total number of anionic charges to the total number of cationic charges of from 95/5 to 5/95, very particularly from 90/10 to 10/90.

Said cationic or amphoteric film-forming homopolymers or copolymers derived from cationic monomers containing ethylenic unsaturation preferably have a molecular mass of less than 100 000 (molecular 5 mass by weight, expressed in g/mol, determinable by aqueous gel permeation chromatography (GPC) or measurement of the viscosity in 1N NaNO₃ solution.

The invention secondly provides a film-forming dispersion comprising
10 - in the order of from 0.01 to 15% of its weight, preferably in the order of from 0.1 to 10% of its weight of titanium dioxide in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is
15 greater than 150 m²/g, preferably greater than 200 mg²/g,
- and in the order of from 0.005 to 15% of its weight, preferably in the order of from 0.01 to 10% of its weight, of at least one film-forming polyalkoxylated
20 organosiloxane or organic polymer,
the continuous phase of said dispersion comprising water and/or at least one alcohol whose boiling point is less than 120°C, preferably less than or equal to 100°C, and having, when it comprises water, a pH
25 different by at least 1 unit, preferably at least 2 units, from the value of the isoelectric point of titanium dioxide in said dispersion.

Preferential or more particular features

regarding the nature of the titanium dioxide, the continuous phase, pH regulators, other additives that may be present, and respective amounts of various components, have already been indicated above.

5 Examples of film-forming alkoxylated organosiloxane or organic polymers which may be employed to improve the film-forming nature of the titanium nanoparticles have already been mentioned above.

10 Said dispersion may be obtained by mixing its various components at ambient temperature.

An especially advantageous film-forming dispersion comprises

- in the order of from 0.01 to 15% of its weight,
15 preferably in the order of from 0.1 to 10% of its weight of titanium dioxide in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is greater than 150 m²/g, preferably greater than 200 m²/g,
20 - and in the order of from 0.005 to 10% of its weight, preferably in the order of from 0.01 to 5%, more particularly from 0.01 to 2% of its weight, of at least one film-forming organic polymer which is preferably hydrophilic and is capable of interacting with the
25 surface of the titanium dioxide particles, preferably by electrostatic bonding,

the continuous phase of said dispersion comprising water and/or at least one alcohol whose boiling point

is less than 120°C, preferably less than or equal to 100°C, and having, when it comprises water, a pH different by at least 1 unit, preferably at least 2 units, from the value of the isoelectric point of 5 titanium dioxide in said dispersion.

They are preferably aqueous and have a pH of the order of from 4 to 9.

Examples of film-forming organic polymers which may be employed for interacting with the surface 10 of the titanium dioxide particles, preferably by electrostatic bonding, have already been stated above.

The invention lastly provides a process for cleaning and/or disinfecting surfaces exposed to light by depositing and then drying on said surfaces a film-forming titanium dioxide dispersion, said titanium dioxide being in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is greater than 150 m²/g, preferably greater than 200 m²/g, the 20 continuous phase of said dispersion comprising water and/or at least one alcohol whose boiling point is lower than 120°C, preferably lower than or equal to 100°C, said dispersion having, when it comprises water, a pH different by at least 1 unit, preferably by at least 2 units, from the value of the isoelectric point 25 of titanium dioxide in the dispersion.

Preferential or more particular features regarding the nature of the titanium dioxide, the

DEPOSITO DE LA INVENCIÓN

continuous phase, pH regulators, other additives that may be present, especially film-forming polymers, and respective amounts of various components, have already been mentioned above.

5 The titanium dioxide nanoparticle dispersion may be deposited on these surfaces by fine spraying onto the surface to be treated, by application with the aid of a sponge, a cloth or with the aid of a preimpregnated cellulosic material, or any other means,
10 in such a way as to deposit in the order of from 0.01 to 10 g of titanium dioxide per m² of the surface to be treated, preferably in the order of from 0.05 to 5 g of titanium dioxide per m² of the surface to be treated.

The examples which follow are given by way of
15 illustration.

Example 1

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm whose specific surface area is 250 m²/g after drying at
20 200°C, in a water/alcohol mixture, whose composition is as follows:

15% dispersion of titanium dioxide (anatase) in ethylene glycol	3%
REPEL O TEX QCJ (15% aqueous dispersion of film-forming antisoiling ethylene terephthalate/polyoxyethylene terephthalate copolymer)	13%
isopropanol	47.5%
deionized water	36.5%

by introducing the film-forming antisoiling copolymer into the dispersion of titanium dioxide in ethylene glycol, followed by addition of the water/isopropanol

- 5 mixture and then adjustment of the pH to 2.5 using hydrochloric acid.

Example 2

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm
 10 whose specific surface area is 250 m²/g after drying at 200°C, in a water/alcohol mixture, whose composition is as follows:

15% colloidal aqueous dispersion of titanium dioxide (anatase)	3%
REPEL O TEX QCJ (15% aqueous dispersion of film-forming antisoiling ethylene terephthalate/polyoxyethylene terephthalate copolymer)	3%
isopropanol	47%
deionized water	47%

by introducing the film-forming antisoiling copolymer into the colloidal aqueous dispersion of titanium dioxide, followed by addition of the water/isopropanol mixture and then adjustment of the pH to 2.5.

Example 3

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm whose specific surface area is 250 m²/g after drying at 10 200°C, in a water/alcohol mixture, whose composition is as follows:

15% colloidal aqueous dispersion of titanium dioxide	25%
REPEL O TEX QCJ (15% aqueous dispersion of film-forming antisoiling ethylene terephthalate/polyoxyethylene terephthalate copolymer)	8.3%
Isopropanol	33.3%
Deionized water	33.4%

by introducing the film-forming antisoiling copolymer into the colloidal aqueous dispersion of titanium dioxide, followed by addition of the water/isopropanol mixture and then adjustment of the pH to 2.5.

5 **Example 4**

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm whose specific surface area is 250 m²/g after drying at 200°C, in a water/alcohol mixture, whose composition is

10 as follows:

15% colloidal aqueous dispersion of titanium dioxide	3%
SILICONE COPOLYOL 10646 (film-forming polyalkoxylated polydimethylsiloxane)	0.5%
isopropanol	47%
deionized water	49.5%

by introducing the film-forming polymer (imparting brightness and oleophobicity) into the colloidal aqueous dispersion of titanium dioxide, followed by addition of the water/isopropanol mixture and then 15 adjustment of the pH to 2.5.

Example 5

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm whose specific surface area is 250 m²/g after drying at 200°C, in a water/alcohol mixture, whose composition is 20 as follows:

15% colloidal aqueous dispersion of titanium dioxide	25%
GLOKILL PQ (50% by weight aqueous solution of film-forming bacteriostatic cationic polymer diluted to 15%)	8.3%
isopropanol	33.3%
deionized water	33.4%

by introducing the bacteriostatic film-forming polymer into the colloidal aqueous dispersion of titanium dioxide, followed by addition of the water/isopropanol mixture and then adjustment of the pH to 2.5.

5 Example 6

A dispersion is prepared of titanium dioxide (anatase) in the form of elementary particles of 40 nm whose specific surface area is 250 m²/g after drying at 200°C, in a water/alcohol mixture, whose composition is 10 as follows:

Reference	A	B
15% colloidal aqueous dispersion of titanium dioxide	3%	3%
GLOKILL PQ (cationic polymer)	0.1%	0%
isopropanol	5%	5%
deionized water	91.9%	92%

The pH of the dispersion is subsequently adjusted by adding sodium hydroxide to pH = 6.5. In the presence of cationic polymer (test A) the dispersion remains homogeneous and the particle size

measurement by laser scattering confirms the absence of aggregation of titanium dioxide (particle size: 40 nm). In the case of the solution without cationic polymer (test B) severe precipitation is observed when sodium hydroxide is added.

The two neutralized solutions A and B are deposited on a black ceramic tile with the aid of adsorbent paper. The amount deposited is of the order of 0.5 mg/cm².

After drying in the open air, the solution A gives a layer of titanium invisible to the eye. In contrast, solution B gives very distinct white marks. Observation by optical microscopy confirms that the polymer allows an effective state of dispersion to be obtained during drying.

A model soil, stearic acid, is subsequently deposited on each ceramic tile which had been treated before and with formulas A and B. The layer of stearic acid is of approximately 0.01 mg/m² and it is obtained by depositing a 1% solution in isopropanol. After drying, the tiles are exposed to visible light for 48 hours.

Following exposure, the ceramic tile treated with solution A has virtually no trace of stearic acid visible to the eye.

The tile treated with solution B exhibits visible traces of stearic acid.

Example 7

On a ceramic tile, a film corresponding to 3 or 6 g of titanium dioxide per m² of tile surface is formed by spreading the dispersion from example 2 over 5 said tile using a film-drawing device.

The film is left to dry in ambient air overnight.

A suspension of bacteria (*Pseudomonas Aeruginosa*) is deposited over the entire surface of the tile.

The tile, covered with a UV-impermeable cover (for the 10 purpose of preventing excessive dehydration of the bacteria), is exposed to UV light (365 nm) for 6 hours.

The number of colonies is counted and compared with that obtained by depositing the same bacteria suspension on an untreated tile.

15 The results obtained are as follows:

Sample	Titanium dioxide deposited (g/m ²)	Number of colonies (cfu/ml)
Untreated tile	-	5×10^7
Treated tile	3	< 10
Treated tile	6	< 10

CLAIMS

1) The use of a titanium dioxide dispersion for cleaning and/or disinfecting surfaces exposed to light by deposition of a film of titanium dioxide on 5 said surfaces, said titanium dioxide being in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is greater than 150 m²/g, preferably greater than 200 m²/g, the continuous phase of said dispersion 10 comprising water and/or at least one alcohol whose boiling point is less than 120°C, preferably less than or equal to 100°C, said dispersion having, when it comprises water, a pH different by at least 1 unit, preferably by at least 2 units, from the value of the 15 isoelectric point of titanium dioxide in said dispersion.

2) The use as claimed in claim 1), characterized in that the titanium dioxide is in the form of elementary particles whose size is of the order 20 of from 20 to 60 nm and whose specific surface area is of the order of from 200 to 300 m²/g.

3) The use as claimed in claim 1) or 2), characterized in that the elementary particles of titanium dioxide are anatase.

25 4) The use as claimed in any one of claims 1) to 3), characterized in that said dispersion comprises in the order of from 0.01 to 15% of its weight, preferably in the order of from 0.1 to 10% of

DOCUMENT NUMBER

its weight, of titanium dioxide.

5) The use as claimed in any one of claims 1) to 4), characterized in that the alcohols which may constitute or be present in the continuous phase are aliphatic monoalcohols whose boiling point is less than 100°C.

6) The use as claimed in any one of claims 1) to 5), characterized in that the continuous phase comprises water.

10 7) The use as claimed in claim 6) characterized in that the continuous phase comprises from 50 to 100%, preferably from 70 to 99.9% of water.

8) The use as claimed in claim 6) or 7) characterized in that the dispersion has a pH of from 0 15 to 14, preferably from 2 to 14.

9) The use as claimed in claim 8), characterized in that the pH of the dispersion is situated at a value below 4 or above 8, preferably at a value of from 0 to 3 or from 9 to 14, when said 20 dispersion is devoid of additive capable of modifying the isoelectric point of titanium dioxide.

10) The use as claimed in any one of claims 1) to 8), characterized in that said dispersion further comprises at least one film-forming organic or 25 organosiloxane polymer.

11) The use as claimed in claim 10), characterized in that the amount of film-forming polymer represents in the order of from 0.005 to 15%,

preferably in the order of from 0.01 to 10% of said dispersion.

- 12) The use as claimed in claim 10) or 11), characterized in that said film-forming polymers are
- 5 - optionally sulfonated terephthalic oligoesters or copolyesters
- polyoxyalkylenated polymers which carry anionic functions
- cationic or amphoteric polymers
- 10 - polyalkoxylated polydimethylsiloxanes
- film-forming polymers derived from ethylenically unsaturated monomers polymerizable by radical means.

- 13) The use as claimed in claim 12) characterized in that said polymer is capable of
- 15 interacting with the surface of the titanium dioxide particles by electrostatic bonding.

- 14) The use as claimed in claim 13)
- characterized in that said film-forming polymer is
- a sulfonated terephthalic oligoester or copolyester
- 20 - a polyoxyalkylenated polymer which carries anionic functions, such as copolymers of acrylic acid and/or methacrylic acid and polyethylene glycol acrylate and/or methacrylate whose polyoxyethylene unit has a mass of from 500 to 10 000
- 25 - a cationic polymer additionally exhibiting bacteriostatic properties, such as
- quaternary ammonium ionenes
 - cationic polymers derived from epichlorohydrin and

dimethylamine and those derived from epichlorohydrin and imidazole

- a cationic homopolymer or copolymer derived from at least one cationic monomer containing ethylenic

5 unsaturation: a monomer such as

* aminoacryloyl or -acryloyloxy monomers such as

trimethylaminopropyl methacrylate chloride,

trimethylaminoethylacrylamide or -methacrylamide

chloride or bromide, trimethylaminobutylacrylamide or

10 -methacrylamide methyl sulfate, trimethylaminopropyl-methacrylamide methyl sulfate (MES),

(3-methacrylamidopropyl)trimethylammonium chloride

(MAPTAC), (3-acrylamidopropyl)trimethylammonium

chloride (APTAC), methacryloyloxyethyl

15 trimethylammonium chloride or methyl sulfate, and acryloyloxyethyltrimethylammonium chloride

* 1-ethyl-2-vinylpyridinium bromide, chloride or methyl sulfate

* N,N-dialkyldiallylamine monomers such as

20 N,N-dimethyldiallylammmonium chloride (DADMAC)

* polyquaternary monomers such as

dimethylaminopropylmethacrylamide chloride,

N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT)

optionally in a mixture with at least one nonionic

25 monomer such as acrylamide, N-isopropylacrylamide,

N,N-dimethylacrylamide, dimethylaminooethylmethacrylate

(DMAEMA), dimethylaminopropylmethacrylamide, vinyl

alcohol, alkyl or hydroxalkyl acrylates or

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methacrylates, polyoxyalkylene glycol acrylates or methacrylates

- an amphoteric copolymer derived from at least one cationic monomer containing ethylenic unsaturation, a

5 monomer like those mentioned above, and at least one anionic monomer containing ethylenic unsaturation, such as

* acrylic, methacrylic, fumaric, maleic, itaconic, N-methacroylalanine, N-acryloylhydroxyglycine acids or
10 anhydrides or their water-soluble salts

* water-soluble sulfonated or phosphonated ethylenically unsaturated monomers, such as sulfopropyl acrylate or its water-soluble salts, water-soluble styrenesulfonates, vinylsulfonic acid and its water-
15 soluble salts or vinylphosphonic acid and its water-soluble salts

optionally in a mixture with at least one nonionic monomer such as acrylamide, N-isopropylacrylamide, N,N-dimethylacrylamide, dimethylaminoethylmethacrylate
20 (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohol, alkyl or hydroxyalkyl acrylates or methacrylates, polyoxyalkylene glycol acrylates or methacrylates.

15) The use as claimed in claim 14)
25 characterized in that said cationic or amphoteric polymer derived from at least one cationic monomer containing ethylenic unsaturation is
* a DIQUAT homopolymer

- * a DADMAC homopolymer
- * a MAPTAC/acrylic or methacrylic acid; DIQUAT/acrylic or methacrylic acid; DADMAC/acrylic or methacrylic acid copolymer
- 5 * a MES/acrylic or methacrylic acid/DMAEMA copolymer
- * a MAPTAC/acrylic acid/acrylamide; MAPTAC/maleic anhydride/acrylamide; MAPTAC/vinylsulfonic acid/acrylamide copolymer
- * a DADMAC/acrylic acid/acrylamide; DADMAC/maleic anhydride/acrylamide; DADMAC/vinylsulfonic acid/acrylamide copolymer
- 10 * a DIQUAT/acrylic acid/acrylamide; DIQUAT/maleic anhydride/acrylamide; DIQUAT/vinylsulfonic acid/acrylamide copolymer,
- 15 said copolymer having a ratio of the total number of anionic charges to the total number of cationic charges of from 95/5 to 5/95, very particularly from 90/10 to 10/90.
- 16) The use as claimed in any one of
- 20 claims 13) to 15), characterized in that said dispersion comprises from 0.005 to 10%, preferably from 0.01 to 5%, very particularly from 0.01 to 2% by weight of said film-forming polymer capable of interacting with the surface of the titanium dioxide particles by
- 25 electrostatic bonding.

17) The use as claimed in any one of claims 13) to 16), characterized in that said dispersion comprises water and has a pH of from 4 to 9.

18) A film-forming dispersion comprising
- from 0.01 to 15% of its weight, preferably from 0.1
to 10% of its weight of titanium dioxide in the form of
elementary particles whose size is less than 100 nm,
5 preferably less than 70 nm, and whose specific surface
area is greater than 150 m²/g, preferably greater than
200 m²/g,
- and from 0.005 to 15% of its weight, preferably of
the order of from 0.01 to 10% of its weight, of at
10 least one film-forming polyalkoxylated organosiloxane
or organic polymer,
the continuous phase of said dispersion comprising
water and/or at least one alcohol whose boiling point
is less than 120°C, preferably less than or equal to
15 100°C, and having, when it comprises water, a pH
different by at least 1 unit, preferably at least
2 units, from the value of the isoelectric point of
titanium dioxide in said dispersion.

19) The dispersion as claimed in claim 18),
20 characterized in that the titanium dioxide is in the
form of elementary particles whose size is of the order
of from 20 to 60 nm and whose specific surface area is
of the order of from 200 to 300 m²/g.

20) The dispersion as claimed in claim 18)
25 or 19), characterized in that the elementary particles
of titanium dioxide are anatase.

21) The dispersion as claimed in any one of
claims 18) to 20), characterized in that the alcohols

which may constitute or be present in the continuous phase are aliphatic monoalcohols whose boiling point is less than 100°C.

22) The dispersion as claimed in any one of
5 claims 18) to 21), characterized in that the continuous phase comprises water.

23) The dispersion as claimed in claim 22)
characterized in that the continuous phase comprises from 50 to 100%, preferably from 70 to 99.9% of water.

10 24) The dispersion as claimed in claim 22)
or 23) characterized in that the dispersion has a pH of from 0 to 14, preferably from 2 to 14.

25) The dispersion as claimed in any one of
claims 18) to 24), characterized in that said film-
15 forming polymers are

- optionally sulfonated terephthalic oligoesters or copolyesters
- polyoxyalkylenated polymers which carry anionic functions
- cationic or amphoteric polymers
- polyalkoxylated polydimethylsiloxanes
- film-forming polymers derived from ethylenically unsaturated monomers polymerizable by radical means.

26) The dispersion as claimed in claim 25)
25 characterized in that said polymer is capable of interacting with the surface of the titanium dioxide particles by electrostatic bonding.

27) The dispersion as claimed in claim 26)

characterized in that said film-forming polymer is

- a sulfonated terephthalic oligoester or copolyester

- a polyoxyalkylenated polymer which carries anionic functions, such as copolymers of acrylic acid and/or

5 methacrylic acid and polyethylene glycol acrylate

and/or methacrylate whose polyoxyethylene unit has a mass of from 500 to 10 000

- a cationic polymer additionally exhibiting

bacteriostatic properties, such as

10 • quaternary ammonium ionenes

• cationic polymers derived from epichlorohydrin and dimethylamine and those derived from epichlorohydrin and imidazole

- a cationic homopolymer or copolymer derived from

15 at least one cationic monomer containing ethylenic unsaturation: a monomer such as

* aminoacryloyl or -acryloyloxy monomers such as

trimethylaminopropyl methacrylate chloride,

trimethylaminoethylacrylamide or -methacrylamide

20 chloride or bromide, trimethylaminobutylacrylamide or -methacrylamide methyl sulfate, trimethylaminopropyl-methacrylamide methyl sulfate (MES),

(3-methacrylamidopropyl)trimethylammonium chloride

(MAPTAC), (3-acrylamidopropyl)trimethylammonium

25 chloride (APTAC), methacryloyloxyethyl

trimethylammonium chloride or methyl sulfate, and

acryloyloxyethyltrimethylammonium chloride

* 1-ethyl-2-vinylpyridinium bromide, chloride or methyl

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sulfate

- * N,N-dialkyldiallylamine monomers such as
N,N-dimethyldiallylammonium chloride (DADMAC)
- * polyquaternary monomers such as
- 5 dimethylaminopropylmethacrylamide chloride,
N-(3-chloro-2-hydroxypropyl)trimethylammonium (DIQUAT)
optionally in a mixture with at least one nonionic
monomer such as acrylamide, N-isopropylacrylamide,
N,N-dimethylacrylamide, dimethylaminoethylmethacrylate
- 10 (DMAEMA), dimethylaminopropylmethacrylamide, vinyl
alcohol, alkyl or hydroxyalkyl acrylates or
methacrylates, polyoxyalkylene glycol acrylates or
methacrylates
 - an amphoteric copolymer derived from at least one
- 15 cationic monomer containing ethylenic unsaturation, a
monomer like those mentioned above, and at least one
anionic monomer containing ethylenic unsaturation, such
as
 - * acrylic, methacrylic, fumaric, maleic, itaconic,
- 20 N-methacroylalanine, N-acryloylhydroxyglycine acids or
anhydrides or their water-soluble salts
 - * water-soluble sulfonated or phosphonated
ethylenically unsaturated monomers, such as sulfopropyl
acrylate or its water-soluble salts, water-soluble
- 25 styrene sulfonates, vinylsulfonic acid and its water-
soluble salts or vinylphosphonic acid and its water-
soluble salts
 - optionally in a mixture with at least one nonionic

monomer such as acrylamide, N-isopropylacrylamide, N,N-dimethylacrylamide, dimethylaminoethylmethacrylate (DMAEMA), dimethylaminopropylmethacrylamide, vinyl alcohol, alkyl or hydroxyalkyl acrylates or 5 methacrylates, polyoxyalkylene glycol acrylates or methacrylates.

- 28) The dispersion as claimed in claim 27) characterized in that said cationic or amphoteric polymer derived from at least one cationic monomer 10 containing ethylenic unsaturation is
- * a DIQUAT homopolymer
 - * a DADMAC homopolymer
 - * a MAPTAC/acrylic or methacrylic acid; DIQUAT/acrylic or methacrylic acid; DADMAC/acrylic or methacrylic acid 15 copolymer
 - * a MES/acrylic or methacrylic acid/DMAEMA copolymer
 - * a MAPTAC/acrylic acid/acrylamide; MAPTAC/maleic anhydride/acrylamide; MAPTAC/vinylsulfonic acid/acrylamide copolymer
 - * a DADMAC/acrylic acid/acrylamide; DADMAC/maleic anhydride/acrylamide; DADMAC/vinylsulfonic acid/acrylamide copolymer 20
 - * a DIQUAT/acrylic acid/acrylamide; DIQUAT/maleic anhydride/acrylamide; DIQUAT/vinylsulfonic acid/acrylamide copolymer
 - 25 acid/acrylamide copolymer,
said copolymer having a ratio of the total number of anionic charges to the total number of cationic charges of from 95/5 to 5/95, very particularly from 90/10 to

10/90.

29) The dispersion as claimed in any one of claims 26) to 28), characterized in that said dispersion comprises from 0.005 to 10%, preferably from 5 0.01 to 5%, very particularly from 0.01 to 2% by weight of said film-forming polymer capable of interacting with the surface of the titanium dioxide particles by electrostatic bonding.

30) The dispersion as claimed in any one of 10 claims 26) to 29), characterized in that said dispersion comprises water and has a pH of from 4 to 9.

31) A process for cleaning and/or disinfecting surfaces exposed to light by depositing and then drying on said surfaces a film-forming 15 titanium dioxide dispersion, said titanium dioxide being in the form of elementary particles whose size is less than 100 nm, preferably less than 70 nm, and whose specific surface area is greater than 150 m²/g, preferably greater than 200 m²/g, the continuous phase 20 of said dispersion comprising water and/or at least one alcohol whose boiling point is lower than 120°C, preferably lower than or equal to 100°C, said dispersion having, when it comprises water, a pH different by at least 1 unit, preferably by at least 25 2 units, from the value of the isoelectric point of the titanium dioxide in said dispersion.

32) The process as claimed in claim 31), characterized in that the amount of titanium dioxide

deposited on the hard surfaces is of the order of from 0.01 to 10 g of titanium dioxide per m² of the surface to be treated, preferably of the order of from 0.05 to 5 g of titanium dioxide per m² of the surface to be
5 treated.

33) The process for cleaning and/or disinfecting as claimed in claim 31) or 32), by deposition of the film-forming dispersion used as claimed in any one of claims 1) to 17) or claimed in
10 any one of claims 18) to 30).

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COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(include Reference to PCT International Applications) **PCT/FR00/00371**

ATTORNEY'S DOCKET NO
RN99013

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

USE OF FILM-FORMING TITANIUM DIOXIDE DISPERSIONS FOR CLEANING AND DISINFECTION SURFACES, FILM-FORMING TITANIUM DIOXIDE DISPERSIONS

the specification of which (check only one item below):

- is attached hereto.
 was filed as United States application

Serial No. _____

on _____

and was amended

on _____ (if applicable)

- was filed as PCT international application

Number **PCT/ FR00/00371**

on **February 15, 2000**

and amended under PCT ARTICLE 19

on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations. §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:

COUNTRY PCT indicate PCT	APPLICATION NUMBER	DATE OF FILING (day month year)	PRIORITY CLAIMED UNDER 35 USC 119	
FRANCE	99/01938	17 February 1999	<input checked="" type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO
			<input type="checkbox"/> YES	<input type="checkbox"/> NO

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONTINUED) include Reference to PCT International Applications) PCT/FR00/00371				ATTORNEY'S DOCKET NO RN99013
<p>I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations. §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:</p>				
PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120				
U.S. APPLICATIONS		STATUS (CHECK ONE)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED
PCT APPLICATIONS DESIGNATING THE U.S.				
PCT APPLICATION NO	PCT FILING DATE	US SERIAL NUMBERS ASSIGNED (if any)		
<p>POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney's and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (List name and registration number)</p> <p>JOHN A. SHEDDEN - Reg. No. 25,644, Kevin McVEIGH - Reg. No. 33017 JOHN D. WOOD - Reg. No. 31,146</p>				
<p>Send Correspondence to: <u>John Daniel WOOD</u> <u>INTELLECTUAL PROPERTY DEPT.</u> <u>RHODIA INC.</u> <u>259 PROSPECT PLAINS ROAD,</u> <u>CN 7500,</u> <u>CRANBURY, NJ 08512-7500</u></p>			<p>Direct Telephone Calls to: (name and telephone number) JOHN D. WOOD (609) 860-4192</p>	
201	FULL NAME OF INVENTOR	FAMILY NAME <u>AUBAY</u>	FIRST GIVEN <u>Eric</u>	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY <u>LE PERREUX SUR MARNE</u>	STATE OR FOREIGN COUNTRY <u>FRANCE</u>	COUNTRY OF CITIZENSHIP <u>FRANCE</u>
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202	FULL NAME OF INVENTOR	FAMILY NAME <u>CHOPIN</u>	FIRST GIVEN <u>Thierry</u>	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY <u>SAINT-LEU LA FORET</u>	STATE OR FOREIGN COUNTRY <u>FRANCE</u>	COUNTRY OF CITIZENSHIP <u>FRANCE</u>
	POST OFFICE ADDRESS	6, rue Diderot	CITY <u>SAINT-LEU LA FORET</u>	STATE & ZIP CODE/COUNTRY <u>F-95320, FRANCE</u>
203	FULL NAME OF INVENTOR	FAMILY NAME <u>GEFFROY</u>	FIRST GIVEN <u>Cédric</u>	SECOND GIVEN NAME
	RESIDENCE & CITIZENSHIP	CITY <u>PARIS</u>	STATE OR FOREIGN COUNTRY <u>FRANCE</u>	COUNTRY OF CITIZENSHIP <u>FRANCE</u>
	POST OFFICE ADDRESS	4A, Rue Marmontel	CITY <u>PARIS</u>	STATE & ZIP CODE/COUNTRY <u>F-75015, FRANCE</u>
<p>I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true: and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.</p>				
SIGNATURE OF INVENTOR 201	SIGNATURE OF INVENTOR 202		SIGNATURE OF INVENTOR 203	
<u>C. -</u>	<u>Chopin</u>		<u>Geffroy</u>	
DATE <u>August 1 2001</u>	DATE <u>7th of August 2001</u>		DATE <u>6th of August 2001</u>	

**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY
(CONTINUED) include Reference to PCT International Applications** PCT/FR00/00371

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RN99013

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations. §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

PRIOR U.S. APPLICATIONS OR PCT INTERNATIONAL APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. 120

U.S. APPLICATIONS			STATUS (CHECK ONE)		
U.S. APPLICATION NUMBER	U.S. FILING DATE		PATENTED	PENDING	ABANDONED
PCT APPLICATIONS DESIGNATING THE U.S.					
PCT APPLICATION NO	PCT FILING DATE	US SERIAL NUMBERS ASSIGNED (if any)			

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney's and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (List name and registration number) JOHN A. SHEDDEN - Reg. No. 25,644,
Kevin McVEIGH - Reg. No. 33017
JOHN D. WOOD - Reg. No. 31,146

Send Correspondence to: John Daniel WOOD INTELLECTUAL PROPERTY DEPT. RHODIA INC. 259 PROSPECT PLAINS ROAD, CN 7500, CRANBURY, NJ 08512-7500	Direct Telephone Calls to: (name and telephone number) JOHN D. WOOD (609) 860-4192
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204	FULL NAME OF INVENTOR	FAMILY NAME GUILLOU	FIRST GIVEN NAME Véronique	SECOND GIVEN NAME
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205	FULL NAME OF INVENTOR	FAMILY NAME LEHAUT	FIRST GIVEN NAME Corinne	SECOND GIVEN NAME
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206	FULL NAME OF INVENTOR	FAMILY NAME	FIRST GIVEN NAME	SECOND GIVEN NAME
RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP	
POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE & ZIP CODE/COUNTRY	

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 204 <i>V. Guillou</i>	SIGNATURE OF INVENTOR 205 <i>C. Leaut</i>	SIGNATURE OF INVENTOR 206
DATE <i>22 of August 2001</i>	DATE <i>02 August 2001</i>	DATE